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# CHARACTERIZATION OF CERAMIC COMPOSITE MATERIAL USING TERAHERTZ REFLECTION IMAGING TECHNIQUE (PREPRINT)

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# Characterization of Ceramic Composite Materials using Terahertz Reflection Imaging Technique.

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Abstract—THz time-domain reflection imaging is performed to do non-destructive evaluation on ceramic composite materials in order to characterize changes in material properties due to mechanical and thermal degradation.

#### I. INTRODUCTION AND BACKGROUND

The characterization of defects such as rust, voids, etc. on materials and the analysis and prediction of strain and stress induced breakdown are well known applications of non-destructive evaluation (NDE) techniques. The use of terahertz (THz) radiation as an analysis and monitoring tool for novel materials has increased rapidly in recent years. [11-3] While suggested as a potential NDE tool for use in the field of ceramic and ceramic matrix composite materials, the use of THz spectroscopy and imaging in the examination of the effects of mechanical and thermally induced degradation of ceramic composite materials is not well established.

THz time-domain images were acquired using a commercial system manufactured by Teraview. Ultrafast laser pulses with an 800 nm center wavelength and 100 fs pulse width triggered a fiber-coupled GaAs photoconductive antenna (PCA). Collimated THz light from the PC antenna transmitter was focused via a 50 mm focal length lens (f# = 2) onto the samples at a near-normal incident angle. The reflected radiation was detected by a PCA receiver module, based on LT-GaAs, with an identical lens configuration. When the system is optimized and calibrated using a metal sample target, the typical bandwidth of a detected THz pulse exceeds 3 THz.

Tests were conducted on both oxide-based and silicon nitride carbon based ceramic matrix composite (CMC) samples. A 5.9 x 17.4 cm area is scanned, containing both an aluminum reference and the CMC sample. A full time-domain waveform, 250 ps long, is acquired for each 0.5 x 0.5 mm pixel. For this comprehensive study, there are multiple rounds of data acquisition. The first round consists of spectroscopic imaging of all of the untreated samples. Additional rounds will consist of imaging of all the samples following treatments of either thermally or mechanically induced degradation of varying magnitudes and also combinations of the two differing types of treatments. Comparison of the data both in the timedomain and frequency-domain acquired from this imaging of both the untreated and treated samples assesses whether or not the magnitude and extent of the induced changes can be monitored using terahertz imaging and spectroscopy.

## II. RESULTS

In order to analyze the acquired data, a program was written in MATLAB that generates images based on the maximum

amplitude of the time-domain pulse as well as changes in the arrival time of the pulse (time of flight). Images are also generated based on the electric field amplitude at specified frequencies and frequency weighted reflectivity. In this latter case, the amplitude of each pixel is based on the integration of the full spectral content of the detected pulse. Fig. 1 is an image based on the frequency weighted reflectivity from the A-29 Oxide Sample prior to any stress treatment. Note that the textured weave on the surface of the sample is clearly visible. Quantitative analysis is also available in the program producing graphs of normalized reflectivity (averaged over the entirety of the sample) as a function of frequency. In all cases, the reflectivity is based on comparison of a THz pulse reflected from a CMC sample compared to the aluminum reference.

The analysis described above operates under the assumption that all stress-induced changes in the CMC samples are uniform across the material. Consequently, we are also examining changes in the reflectivity of these samples preand post- stress treatment over smaller spatial sub-domains. A grid system has been developed that allows for point by point analysis of the imaging data. As a result, it is possible to compare specific spatial points, with a resolution less than 1 mm, on samples prior to and following stress treatment. This analysis approach will assess the spatial variance of any stress induced changes in the terahertz reflectivity.

Fig. 2 shows time domain (TD) pulses obtained from reflection measurements of the aluminum, oxide, and SiNC baseline samples. Fig. 3 compares TD pulses from measurements of an untreated SiNC sample compared to two stress treated samples of the same type at the same specified

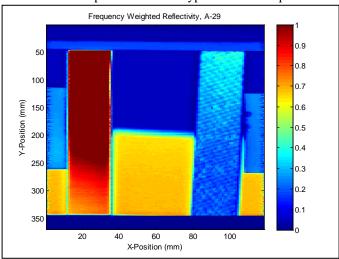


Fig. 1. Frequency Weighted Reflectivity Image of Aluminum Reference Piece (Left) and A-29 Oxide CMC Sample (Right).

spatial location on each sample. Sample B-87, which was heat treated at 1200°C for 100 hours, shows no obvious variation from the baseline measurement. Sample B-60 was fatigue treated under 225 MPa of pressure at 1 Hz for 1000 cycles, and shows attenuation and further signal distortion in the time domain. In comparing the normalized reflectivity (Fig. 4) of these three samples, again the fatigue treated sample exhibits erratic behaviors as compared to the baseline, and there is no obvious difference with the heat treated sample. The erratic reflectivity of the SiNC fatigue-treated sample could be evidence of increased scattering following treatment. Fig. 4 shows the normalized reflectivity from an Oxide sample before and after it underwent a heat treatment of 1000°C for 1000 hours. There is no obvious change in the reflectivity.

In order to validate whether or not THz imaging will be useful in assessing ceramic composite material health, it is necessary to determine if THz spectroscopic imaging can clearly highlight areas of the samples that have been affected by mechanical and thermal treatments. This talk will update progress on efforts to demonstrate characterization of ceramic composite materials using terahertz time-domain reflection imaging.

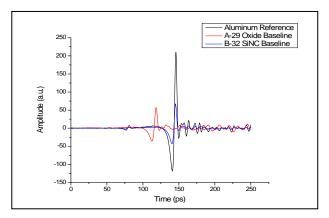


Fig. 2. Time-Domain data from imaging of Aluminum, Oxide, and SiNC baselines.

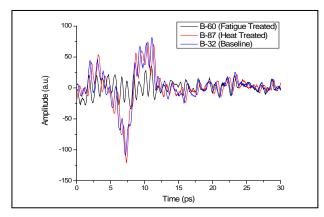


Fig. 3. Time-Domain data from imaging of the SiNC Samples.

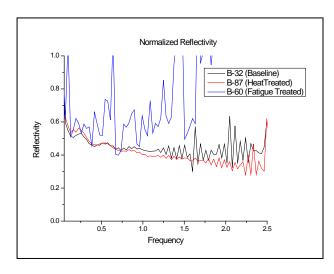


Fig.4. Normalized Reflectivity from untreated and treated SiNC Samples.

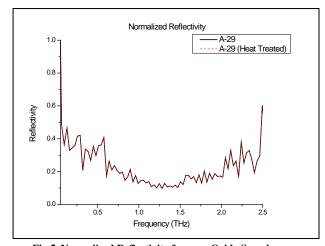


Fig.5. Normalized Reflectivity from an Oxide Samples prior to and after heat treament.

#### REFERENCES

- [1] D. T. Petkie, I. V. Kemp, C. Benton, C. Boyer, L. Owens, J. A. Deibel, C. D. Stoik, M. J. Bohn, "Nondestructive terahertz imaging for aerospace applications," Proceedings of SPIE: Millimetre Wave and Terahertz Sensors and Technology, **7485** (Berlin, Germany, 2 September 2009). [7485-13]
- [2] "Imaging with terahertz radiation," Wai Lam Chan, Jason Deibel, and Daniel M. Mittleman, *Reports on Progress in Physics* **70**, 1325-1379 (2007).
- [3] Naftaly M, et al. Investigation of ceramic boron nitride by terahertz time-domain spectroscopy. *J. Eur. Ceram. Soc.* (2010), doi:10.1016/j.jeurceramsoc.2010.04.040